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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 461.3-2013

Replacing YS/T 461.3-2003

**Methods for chemical analysis of lead and zinc bulk
concentrates. Part 3: The determination of sulfur content.
Combustion-neutralization titrimetric method**

混合铅锌精矿化学分析方法 第3部分：硫量的测定燃烧—中和滴定法
(English Translation)

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Table of Contents

- Foreword
- 1 Scope
- 2 Method summary
- 3 Reagents

Foreword

YS/T 461 "Lead and Zinc concentrates - Methods" is divided into 11 parts.

- Part 1. Determination of the amount of lead and zinc content Na₂EDTA precipitation titration;
- Part 2. Determination of iron content Na₂EDTA titration;
- Part 3. Determination of sulfur content of combustion - neutralization titration method;
- Part 4. Determination of arsenic content of iodine titration;
- Part 5. Determination of silicon dioxide content - Molybdenum blue spectrophotometric method;
- Part 6. Determination of the amount of mercury atomic fluorescence spectrometry;
- Part 7. Determination of cadmium content by flame atomic absorption spectrometry;
- Part 8. Determination of copper content - Flame atomic absorption spectrometry;
- Part 9. Determination of the amount of silver by flame atomic absorption spectrometry;
- Part 10. the amount of gold and silver contents - fire assay;
- Part 11. arsenic, cadmium, copper, cobalt, nickel, antimony, bismuth content - Inductively Coupled Plasma - Atomic Emission Spectrometry.

This section YS/T Part 3 461.

This section drafted in accordance with GB/T 1.1-2009 given rules.

The partial replacement of YS/T 461.3-2003 "Lead and Zinc concentrates - Determination of sulfur content of combustion - neutralization titration method" Compared with YS/T 461.3-2003, the main changes are as follows.

- Measurement range from the original "20.00% - 40.00%" to "15.00% - 38.00%";
- Modified schematic 19,20 two notes;

- Complements the buffer in FIG. 11 bottles;
- Sodium hydroxide standard solution change;
- Added "reproducibility" clause, delete the words "to allow poor" clause;
- Added "the test report" requirement.

The non-ferrous metal part by the National Standardization Technical Committee (SAC/TC243) centralized.

YS/T 461 is responsible for drafting. Shenzhen Lingnan Nonferrous Metals Co., Baiyin Nonferrous Metals Corporation, Beijing Institute of Mining and Metallurgy.

This section is responsible for drafting unit. Baiyin Nonferrous Metals Corporation.

Participated in the drafting of this section. Shenzhen Lingnan Nonferrous Metals Co., Ltd. Shaoguan smelter Shenzhen Lingnan Nonferrous Ltd. belongs Fankou, Daye Nonferrous Metals Group Holdings Limited, Western Mining Corporation and Inspection Center, west Xitiesan Branch Mining Corporation, Shaanxi Dongling Smelting Co., Ltd.

1 Scope

This section YS/T 461 specifies the method for determination of Lead and Zinc concentrate sulfur content.

This section applies to the determination of Lead and Zinc concentrate sulfur content. Measurement range. 15.00% - 38.00%.

2 Method summary

Sample stream at high temperatures in the combustion air, will be converted to sulfur dioxide, the absorption and oxidation with hydrogen peroxide to sulfuric acid to methyl red - methylene Blue mixed solution as an indicator, titrated with standard sodium hydroxide solution to the solution turns from purple to bright green is the end, according to the consumption of hydrogen Volume calculation standard titration solution of sodium sulfur content.

When interfere with the determination of fluorine greater than 0.2%.

3 Reagents

Unless otherwise indicated in the analysis using only recognized as analytical grade reagents and distilled or deionized water or equivalent purity.

3.1

sodium hydroxide.

3.2

discoloration silica gel.

3.3

Qianfen (wPb $\geq$ 99.99%).

3.4

copper oxide (powder).

3.5

Hydrogen peroxide (30%).

3.6

sulfate ($\rho = 1.84\text{g/mL}$).

3.7

sulfuric acid (11), pure class distinctions.

3.8

sulfuric acid (119), pure class distinctions.

3.9

nitric acid (13), pure class distinctions.

3.10

potassium permanganate - sodium hydroxide solution. take 3.0g of potassium permanganate dissolved in 100mL of water, 10g of sodium hydroxide, dissolved load Wash cylinders.

3.11

mixed indicator. methylene blue solution (1.6g/L) and methyl red ethanol solution (1.2g/L), before use by mixing equal volumes.

3.12

absorbing hydrogen peroxide solution. 1000mL 50mL solution containing hydrogen peroxide [30% (mass fraction)], add 1mL mixed indicator (3.11), standard titration with sodium hydroxide solution (3.14) and sulfuric acid (3.8) solution was adjusted just was bright green (using now).

Preparation of the base lead sulfate 3.13 reagents. Weigh 20g of lead oxide (3.3) in the 500mL beaker, add 30mL of nitric acid (3.9) is dissolved, After completion of the reaction the suspension was filtered off, 20mL was added sulfuric acid (3.7), with filtration after 2h sedimentation speed quantitative filter paper, washed with distilled water to Neutral, dried in the oven, put a porcelain crucible, burning in a muffle furnace 780 °C 1h, remove the

coolish desiccator. Removed after room temperature After grinding into a mortar, add 780 °C muffle furnace burning 1h removed, placed in a dryer as a reference material.

3.14

sodium hydroxide standard titration solution (approximately 0.1mol/L).

3.14.1

preparation. 7mL sodium hydroxide solution (400g/L) into a 1L plastic cylinder with boiled and cooled distilled water to 1L, and mix.

3.14.2

Calibration. Weigh 0.40g (accurate to 0.0001g) lead sulfate reference reagent (3.13) in the porcelain boat, covering 0.2g ~ 0.3g copper oxide (3.4), according to the analysis method is calibrated at the same time, record the volume of consumption of sodium hydroxide.

According to equation (1) calculate the standard volumetric sodium hydroxide solution for sulfur titration factor.